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AFRPL-TR-68-16

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INHIBITED N_2O_4 ENGINEERING DATA

Third Quarterly Report

H. E. Dubb
A. D. Lev
B. C. Neale

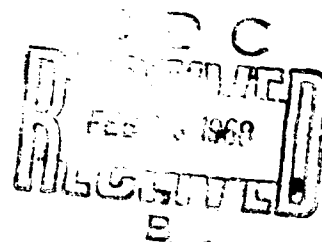
Rocketdyne
A Division of North American Rockwell Corporation
Canoga Park, California

TECHNICAL REPORT AFRPL-TR-68-16

February 1968

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H. E. Dabb
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B. C. Neale

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FOREWORD

The research reported herein was supported by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards Air Force Base, California, Air Force Systems Command, United States Air Force, under Contract F04611-67-C-0099, with Douglas Huxtable, 2nd/Lt/USAF, RPCL, serving as Project Monitor

The work described covers the period 1 November 1967 through 31 January 1968. The Responsible Scientist for this program is Dr. Hubert E. Dubb of Physical and Engineering Chemistry, which is managed by Dr. K. H. Mueller. The work was conducted by members of Physical Chemistry, supervised by Dr. A. E. Axworthy; Engineering Chemistry, supervised by Dr. W. Unterberg; Analytical Chemistry, supervised by Dr. V. H. Dayan; and the Metallurgical Analysis Unit, supervised by Mr. E. F. Green. The principal contributors to the program were Dr. Dubb, Mr. A. D. Lev, and Mr. B. C. Neale.

This report has been assigned the Rocketdyne report No. R-7198-3.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

W. H. EREIKE, Colonel, USAF
Chief, Propellant Division

ABSTRACT

This program is concerned with collecting engineering data to further characterize the new storable oxidizer INTO which is NTO inhibited with 1 to 3 weight percent FNO_2 .

Sensitivity testing of 3 and 9 weight percent solutions of FNO_2 in NTO has been completed. The sensitivity tests employed were detonation propagation, U-tube, and drop weight testing. The mixtures were not sensitive under the test conditions.

All equipment is in-house and has been assembled for stress corrosion testing of 250 maraging steel, 347 stainless steel, and 2219 aluminum alloys exposed to INTO.

Fresh surfaces have been machined in containers of 250 maraging steel, titanium 6Al-4V, A286 steel, 321 stainless steel, nickel, and 6061 aluminum. The containers will be used for studying passivation of these materials against attack by the FNO_2 in INTO.

Atomic absorption methods are being developed to analyze INTO and NTO for dissolved Fe, Cr, Cu, Ni, Ti, and Al. Sensitivities for these six elements dissolved in NTO have been increased twentyfold.

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INTRODUCTION

The use of nitrogen tetroxide (NTO) has been continually hampered by corrosion problems. Dry NTO is not a highly corrosive liquid when in contact with most common metals of construction, but moist NTO is extremely corrosive because of the presence of nitric and nitrous acids formed by the reaction of NTO with water.

It has previously been demonstrated under Contract AF04(611)-10809 (Ref. 1) that the addition of a fluorine oxidizer to NTO leads to a reduction of the nitric and nitrous acid content of the propellant with the concurrent production of HF. It has also been shown (Ref. 1 through 5) that if the fluorine oxidizer is FNO_2 , the resulting oxidizer system is storable at 70 C in passivated containers of aluminum, stainless-steel, nickel, and A286 steel.

The present program is being conducted to obtain further engineering data on INTO, which is NTO containing 1 to 3 weight percent FNO_2 . INTO has been examined for possible sensitivity in the U-tube adiabatic compression, drop weight, and detonation propagation tests. It will be determined at ambient temperature and at 70 C if stress corrosion occurs when 250 maraging steel, 347 stainless steel, and 2219 aluminum are exposed to INTO at 75 percent of their yield strengths. Several methods of passivating metal surfaces against attack by INTO will be investigated. An atomic absorption method is being developed to analyze INTO and NTO for dissolved Fe, Cu, Cr, Ni, Ti, and Al.

TASK I: SHOCK SENSITIVITY

During the past quarter, all sensitivity testing of INTO was completed. Solutions of 3 and 9 weight percent FNO_2 in NTO were found insensitive to all detonation propagation and drop weight tests conducted. U-tube adiabatic compression sensitivity tests were conducted with 7 weight percent FNO_2 (nominal value) in NTO. All U-tube tests were also negative.

EXPERIMENTAL AND DISCUSSION

Detonation Propagation Tests

The experimental procedure for conducting detonation propagation tests with INTO was discussed previously (Ref. 7). During this quarter, three tests were conducted with the following test charges: NTO, 3 weight percent FNO_2 in NTO, and 9 weight percent FNO_2 in NTO. Posttest examination of the charge containers and witness plates showed that the results of all three tests were negative. The charge containers were in approximately the same condition as the container recovered from the calibration test conducted with water. INTO will not propagate a detonation in 1-inch-diameter tubing.

Drop Weight Tests

Drop weight tests were conducted using the standard North American Rockwell open-cup impact tester. The tester is shown schematically in Fig. 1. In the open-cup test, a small (approximately 1/10-inch cube) sample of material to be tested was placed in a 5/8-inch-diameter by 3/8-inch-deep aluminum foil sample cup. The cup was then filled with the test fluid and subjected to impact by a falling weight. Filling and impact was carried out as fast as possible to minimize loss of the highly volatile FNO_2 .

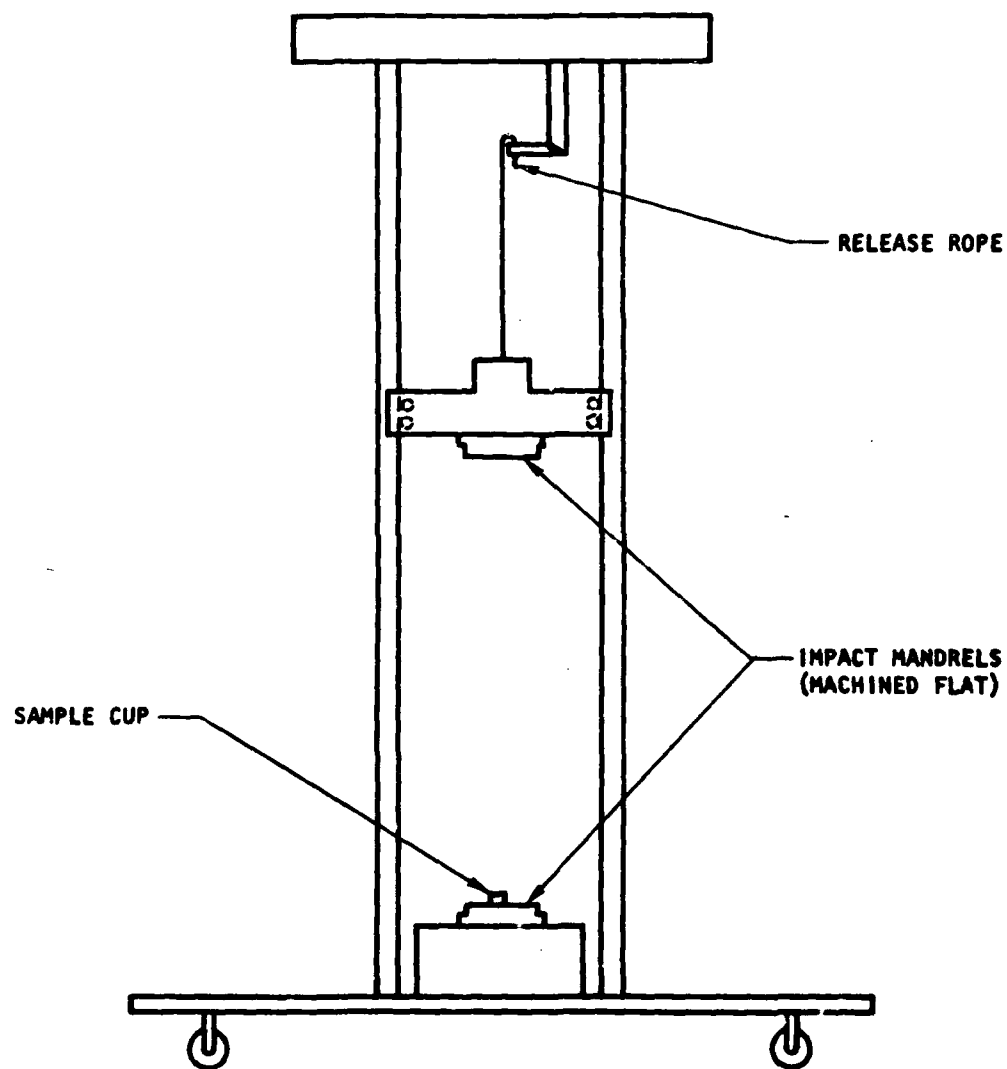


Figure 1. Drop Weight Tester

Impact tests were conducted with both 3 and 9 weight percent FNO_2 solutions (analyses were based on pretest composition of the solutions). The following materials were tested: Teflon, Kynar, Kel-F, phenolic resin-cured butyl rubber, Viton-A, and titanium. All tests were performed at the 68 foot-pound impact level. The 3 weight percent FNO_2 solution was tested at ambient temperature. The 9 weight percent FNO_2 solution was precooled to approximately 15 F for reasons discussed below. Five tests were conducted on each material with each propellant. A control test was conducted with each material using water to determine the posttest condition of the samples due to the impact only. The results of all tests are presented in Table I. It can be seen that all test results were negative, i.e., no aural or visual observation either during or after the test revealed any evidence of combustion or detonation.

TABLE I

DROP WEIGHT TESTS WITH 3 AND 9 WEIGHT
PERCENT FNO_2 IN NTO

Material*	Result	Comments
Butyl Rubber	Negative	Flattening of specimens with blackening of mandrels due to impressment of rubber on mandrels
Viton-A	Negative	More extreme flattening than with butyl rubber but no indication of ignition
Kynar	Negative	Extreme flattening and cracking of specimens
Teflon	Negative	Samples shattered to flakes
Kel-F	Negative	Samples shattered to flakes
Titanium 6Al-4V	Negative	Slight flattening of specimens at edges; samples imbedded in softer mandrels

*Series of five runs with each material for each solution

The loss of FNO_2 under test conditions was estimated from simulated tests. The simulated tests were conducted as follows:

1. A slug of the test solution approximately equal in volume to the test sample was transferred to a 10-milliliter cylinder.
2. The cylinder was conditioned to test temperature (ambient temperature for the 3 weight percent INTO and approximately 15 F for the 9 weight percent INTO).
3. The ullage of the cylinder was opened to the atmosphere for approximately 7 seconds, the time required to load a sample cup and conduct a drop weight test.
4. The liquid in the cylinder was analyzed.

The analysis of the 3 weight percent INTO revealed that the concentration of FNO_2 in the sample cups was actually approximately 0.3 weight percent. For this reason, the 9 weight percent INTO tests were conducted at reduced temperature. It was anticipated that reduced temperature would increase the actual test concentration of FNO_2 . Analysis of the simulated test sample for the 9 weight percent INTO revealed a value of 3 weight percent FNO_2 . Thus, approximately 70 percent of the original FNO_2 was lost to the atmosphere even at 15 F. Since 3 weight percent is the maximum FNO_2 value for INTO (1 to 3 weight percent FNO_2), the second set of drop weight tests are more significant.

U-Tube Tests

The procedure and theory of U-tube adiabatic compression sensitivity testing was discussed previously (Ref. 6). During this reporting period, tests have been conducted with an INTO composition of 7 weight percent FNO_2 in the liquid. Because of condensation difficulties encountered with the 7 weight percent FNO_2 composition, the mode of loading the U-tubes was altered from that used with the 3 weight percent FNO_2 composition (Ref. 6). Instead

of condensing each component separately into the U-tube at liquid nitrogen temperature, a calibrated aliquot of liquid INTO (21 weight percent FNO_2) was dropped directly into the U-tube and then frozen to liquid nitrogen temperature. Because of the large vapor space above the liquid slug in the U-tube, loading of a 21 weight percent mixture results in a liquid composition of 7 weight percent FNO_2 (nominal value) in the U-tube at equilibrium.

Six tests were conducted at 32 F: three with only propellant vapors above the liquid slug and three with 620 mm Hg of air added to the vapor. All tests were conducted at the highest driving pressure available (pressure ratio of approximately 40), and no explosions occurred during any of the tests. INTO, therefore, appears insensitive to this mode of shock stimulus.

CONCLUSIONS

The anticipated insensitivity of INTO has been experimentally verified. With respect to the dangers of shock sensitivity, INTO handling procedures may be essentially those of NT0.

TASK II: STRESS CORROSION

All of the stress corrosion frames, samples, and tanks are in-house. The specimens have been loaded into the frames which have in turn been loaded into the tanks. The tanks have been flushed and filled with dry gaseous nitrogen pending the start of the test. The initiation of the stress corrosion testing is being delayed until the Task III passivation studies are completed.

EXPERIMENTAL

The 2014 aluminum tank, in which the 2219 aluminum specimens are to be stress corrosion tested, has been received and pressure tested. The stress corrosion samples (2219 aluminum, 250 maraging steel, and 347 stainless steel) have been loaded in the stressing frames and the frames have been placed on racks in the tanks. The tanks have been purged with dry gaseous nitrogen and sealed.

FUTURE EFFORT

As soon as the Task III passivation studies are completed, the stress corrosion tanks will be passivated and loaded with INTO.

TASK III: PASSIVATION STUDIES

The original purpose of this task was to study the possible tendency of corrosion products dissolved in INTO to precipitate in small orifices under flow conditions. Results obtained during another program (Ref. 8) have indicated that this phenomenon does not occur with INTO.

Storability tests previously completed (Ref. 2 through 5) have indicated that titanium 6Al-4V and 250 maraging steel are continually attacked by the FNO_2 in INTO when INTO is stored in them after passivation with 20 weight percent solutions of FNO_2 in INTO. Storability tests of INTO in 6061 aluminum, 321 stainless steel, and nickel (Ref. 1) have indicated that the FNO_2 in the INTO does not attack these metals after passivation by gaseous ClF_3 and gaseous F_3NO . Titanium 6Al-4V and 250 maraging steel are ideal materials for use in weight-limited missions because of their high strength-to-weight ratios. Therefore, it is desirable to determine whether INTO can be stored in these alloys after proper passivation. For the preceding reasons, the purpose of this task has been altered to that of studying passivation of metals against attack by the FNO_2 in INTO.

EXPERIMENTAL AND FUTURE EFFORT

The study to be conducted will consist of attempts to passivate A286 steel, titanium 6Al-4V, 250 maraging steel, 6061 aluminum, 321 stainless steel, and nickel by each of three techniques: (1) gaseous FNO_2 - N_2O_4 mixtures, (2) gaseous F_2 , and (3) gaseous ClF_3 . The effectiveness of the passivation techniques will be determined by storing INTO in bombs passivated by each technique and following the FNO_2 content over a 1 to 2 month period. There are not a sufficient number of bombs available to test each of the three passivation techniques simultaneously. Therefore, the bombs will be reamed out at the conclusion of the study of each passivation technique and will be reused. Fresh surfaces have been machined into the bombs preparative to starting the passivation study.

TASK IV: METAL ION ANALYSES

The objective of this Task is the development of methods for the determination of trace amounts of Cr, Cu, Al, Ni, Fe, and Ti in both NTO and INTO utilizing atomic absorption spectrometry.

Efforts during this task have been directed toward increasing the sensitivity of the NTO analyses by evaporative concentration techniques previously reported (Ref. 7). Fourfold and twentyfold concentrations were successfully accomplished. Utilizing this technique, a much greater enhancement of sensitivity is now deemed feasible.

EXPERIMENTAL AND DISCUSSION

NTO Analyses

Fourfold Evaporative Concentration of NTO. Forty milliliters of NTO which had been spiked with Fe, Ni, Cu, Cr, Al, and Ti were carefully evaporated until 10 milliliters of residue remained. The first and third 10-milliliter distillate fractions of the 30 milliliters that were removed were collected in 15-milliliter graduated centrifuge tubes fitted with Fischer-Porter valves. The second 10-milliliter distillate was discarded. The 10-milliliter residue, the two distillate fractions, and an NTO blank were hydrolyzed and analyzed by atomic absorption spectrometric methods previously developed during this program (Ref. 6). Aluminum and titanium analyses were not performed on these samples because at the time these analyses were required, the hood fan was inoperative and it was not considered safe to use the nitrous oxide burner without the proper exhaust.

Significant amounts of the metals were found only in the residue. The data are presented in Table II.

TABLE II

FOURFOLD EVAPORATIVE CONCENTRATION ANALYSIS

Element	Amount Added, micrograms	Percent Recovery		
		Distillate Fraction		Residue
		1	3	
Fe	8	0	0	91
Ni*	2	0	0	91
Cu	8	0	<0**	102
Cr	8	0	0	74

*Inadvertently, 2 instead of 20 micrograms of Ni were added.

**The blank had a slightly higher absorption than this fraction.

The data were examined to determine whether the levels of concentration of the various metals in the hydrolyzed solutions were sufficiently high to provide accurate results. When the NTO residue was hydrolyzed and heated to 90 C to remove the bulk of the nitrous acid, the final volume of the solution was 45 milliliters. Assuming 100 percent recovery in the residue, the concentration of metals in the hydrolyzed residue should be: Cu, 0.18 $\mu\text{g/ml}$; Fe, 0.18 $\mu\text{g/ml}$; Cr, 0.18 $\mu\text{g/ml}$; and Ni, 0.04 $\mu\text{g/ml}$. The detection limits reported by the instrument manufacturer for the four metals are: Cu, 0.005 $\mu\text{g/ml}$; Fe, 0.05 $\mu\text{g/ml}$; Cr, 0.01 $\mu\text{g/ml}$; and Ni, 0.05 $\mu\text{g/ml}$. The detection limit is defined as the concentration of sample in solution giving a signal twice as large as the background variability. It is seen that the sample was doped with slightly less Ni than the literature-reported detection limit for nickel. Therefore, the nickel values are not as accurate as those of the other metals. The levels of the other metals were sufficiently high for accurate results.

The percent recovery of Fe, Cu, and Ni was fairly good at these low concentrations. However, chromium was only recovered to the extent of 74 percent.

This low recovery may be due to chemical depression occurring during the atomic absorption analysis. The instrument manufacturer has reported that chromium is one of several elements that can be chemically depressed. This phenomenon occurs when species are present in the unknown that reduce the absorption of the atomized element, but which are not present in the standards. As previously reported (Ref. 7), there are apparent chemical differences between solutions prepared in 10 percent HNO_3 and those prepared by hydrolyzing NTO to form a HNO_3 solution.

The results of this fourfold concentration by evaporation show good agreement with the results obtained by a previous investigator on this task (Ref. 7) despite the fact that the concentration of the Fe, Cu, and Cr was less by a factor of five and the concentration of the Ni was less by a factor of fifty.

Twentyfold Evaporative Concentration of NTO. One hundred milliliters of NTO which had been spiked with Fe, Ni, Cu, Cr, Al, and Ti were carefully evaporated until a 5-milliliter residue remained. The first 90 milliliters were collected and set aside. The last 5 milliliters of NTO distillate were collected, hydrolyzed, and analyzed along with the 5-milliliter residue. The analysis was conducted in the same manner as described in the fourfold concentration. It was observed that some solid material did not dissolve even after hydrolysis, and therefore some low recoveries were expected. The results are summarized in Table III. They show that the insoluble material was not a major factor in the recovery of the metals.

TABLE III

TWENTYFOLD EVAPORATIVE CONCENTRATION ANALYSIS

Element	Amount Added, micrograms	Percent Recovery	
		5-Milliliter Distillate Prior to Residue	5-Milliliter Residue
Fe	1,000	0	94
Ni	2,000	0	96
Cu	1,000	0	92
Cr	1,000	<0.7	89
Al	700	3	87
Ti	600	0	94

As indicated in Table III, aluminum, and chromium to a lesser extent, were the only metals which appeared in the distillate. The detection limits reported by the instrument manufacturer for these two metals are: aluminum, $0.5 \mu\text{g/ml}$ and chromium, $0.01 \mu\text{g/ml}$. Since the final volume of the hydrolyzed distillate was 41 milliliters, the 3 percent (21 micrograms) aluminum detected in the distillate is equal to its detection limit and may therefore be considered within experimental error. The 0.7 percent (7 micrograms) Cr in the distillate resulted in a solution which was an order of magnitude above the detection limit in the hydrolyzate (0.7 percent chromium represents $0.2 \mu\text{g/ml}$). Thus it seems possible that a small portion of the chromium may have been carried over into the distillate.

The recovery of titanium in the residue was 94 percent. The 6 percent "loss" is below the detection limit for titanium in the hydrolyzed solution. Six percent (36 micrograms) of the titanium is the equivalent of $0.5 \mu\text{g/ml}$ in the hydrolyzed solution. The detection limit for titanium as reported by the instrument manufacturer is $1.0 \mu\text{g/ml}$. Therefore, the recovery of titanium is within experimental error.

The recovery of Fe, Ni, Cu, Cr, and Al are 94, 96, 92, 89, and 87 percent, respectively. The "losses" of these five metals are all above the order of magnitude of the detection limits of these elements in the hydrolyzed solution. As previously mentioned, a small amount of the doping mixture remained in the centrifuge tube, undissolved. This may well account for these small "losses". From the previous results, it can be concluded that a twentyfold concentration of metals in NTO has been successfully demonstrated by evaporation techniques.

FUTURE EFFORT

It is planned to return to the investigation of methods for the analysis of metals in INTO. An attempt will be made to determine the reason for the detection of excessive quantities of Fe, Ni, and Cr in the INTO samples as reported previously (Ref. 7).

SUMMARY

TASK I

Sensitivity testing of INTO has been completed. Solutions of 3 and 8 weight percent FNO_2 in NTO were found to be insensitive in all detonation propagation, drop weight, and U-tube adiabatic compression tests.

TASK II

All of the stress corrosion frames, samples, and tanks are in-house. The specimens have been loaded into the frames and the frame/specimen combinations have been placed in the tanks. The stress corrosion testing will be initiated as soon as the Task III passivation studies are completed.

TASK III

Containers of A286 steel, titanium 6Al-4V, 250 maraging steel, 6061 aluminum, 321 stainless steel, and nickel have been prepared for passivation studies.

TASK IV

Atomic absorption methods are being developed to analyze INTO and NTO for dissolved metals. Fourfold and twentyfold evaporative concentrations of dissolved Cr, Cu, Al, Ni, Fe, and Ti in N_2O_4 have been accomplished. Utilizing the techniques developed, a great enhancement of sensitivity is now deemed feasible.

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